Chemical Standards for Optoelectronic Semiconductors

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Optoelectronic device makers require accurate data on the chemical composition of III-V semiconductor alloys, such as $Al_xGa_{1-x}As$, to make their manufacturing processes more predictable and robust and to enable more accurate modeling and simulation of device performance. As part of a larger NIST project to develop standard reference materials to meet this need, we are improving the accuracy of room-temperature photoluminescence spectroscopy as an indirect, standards-based composition measurement method.

The objective of this project is to improve the accuracy of room-temperature photoluminescence (PL) spectroscopy as a chemical composition measurement method for III-V compound semiconductor films, focusing on the important $Al_xGa_{1-x}As$ and $In_xGa_{1-x}As_yP_{1-y}$ systems. PL is widely used by the semiconductor materials and device industries as a composition metrology tool because of its speed, simplicity, and low cost. However, to achieve the high accuracy needed for semiconductor device simulation and modeling, the PL composition measurements must be calibrated using standard samples of accurately known composition. Heretofore, such samples have not been available.

The Compound Semiconductor Composition Standards Program at NIST is aimed at the development of high-accuracy composition standards for III-V compound semiconductor films. Thus far, our efforts have focused on the $Al_xGa_{1-x}As$ system. Films are grown in two molecular beam epitaxy (MBE) deposition systems and the compositions (parameter x) are measured both by in-situ monitoring, primarily reflection high-energy electron diffraction (RHEED), and by ex-situ chemical measurements, primarily wavelength dispersive x-ray spectroscopy in an electron probe microanalyzer (WDS/EPMA).

The PL-based composition measurements rely on the linear or nearly linear composition (x) dependence of the energy at the peak of the PL emission spectrum, which is closely tied to the semiconductor band gap. Careful data reduction and analysis techniques, including near-real-time calibration of the wavelength (or photon energy) scale with atomic vapor lamps, correction for the wavelength dependence of the spectrometer response function, and curve-fitting of the emission spectrum lineshape, enable us to determine the peak PL emission energy, denoted E_{PL}, with high accuracy and reproducibility. A platinum resistive temperature sensor is used to measure and correct for the effect of room temperature drift on E_{PL}. With these procedures, the reproducibility of E_{PL} is found to be ±0.0003 eV or better (note all uncertainties are reported at the 2σ level).

Measurements of E_{PL} were made for a number of samples with compositions (x) previously measured by the in-situ RHEED and ex-situ WDS methods. Calibration curves were generated by fitting a linear function to the (E_{PL}, x) data. The best-fit line determined from the WDS compositions is

$$x = (0.721\pm0.006\ \mathrm{eV}^{-1})\,[E_{PL} - (1.4232\pm0.0003)\ \mathrm{eV}]$$

The deviations between the fitted PL compositions and the measured WDS compositions, as well as the corresponding deviations for PL vs. RHEED, are plotted in Fig 1. The deviations between the fitted line and the measured compositions are seen to be larger for (E_{PL}, x_{RHEED}) than for (E_{PL}, x_{WDS}). This result is plausible because the RHEED measurements are performed at the start of each deposition, and do not allow for possible composition drift during growth, while both the PL and WDS/EPMA measurements probe the upper portions of the as-grown films.

We will also examine heavily n-type or p-type doped samples, in which large doping shifts of E_{PL} occur. The objective will be to develop an algorithm to determine x from E_{PL} and the dopant (or carrier) concentration.

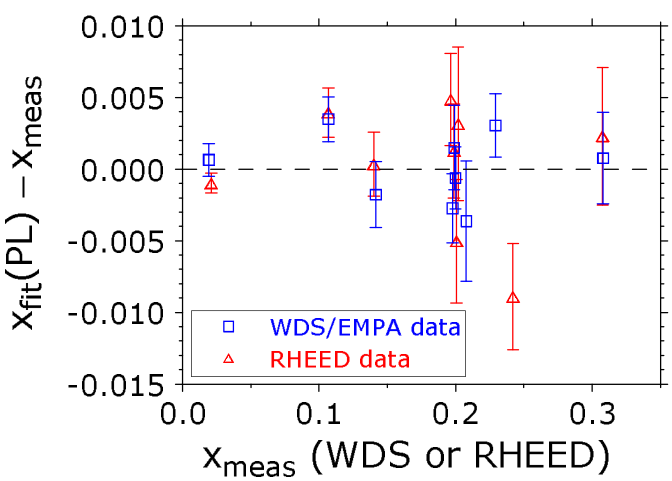

Fig. 1. Deviations of fitted (PL) from measured (WDS, RHEED) compositions of $Al_xGa_{1-x}As$ films. Uncertainties of the fitted PL compositions are derived from the uncertainties of the slope and the E_{PL} values (as given in the equation). The uncertainties of the WDS compositions are estimated to be ≈0.002; the uncertainties of the RHEED compositions are estimated to be ≤0.004.

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